AN ALL-GLASS NITROGEN APPARATUS

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The necessity of refined analytical methods is apparent to any one who has given serious consideration to the problem or problems in certain phases of plant metabolism. This need is especially felt in the problems of nitrogen fixation by the lower forms of plant life. Helpful suggestions may be obtained from the nitrogen methods used in the field of animal biochemistry, yet these methods are usually not directly applicable without more or less revision with a view to obtaining greater accuracy even at the expense of convenience and ease of manipulation. This is illustrated, for instance, by the nitrogen method proposed by Davis[1] while working on the problem of nitrogen fixation by fungi in the laboratory of Professor Duggar. This method may be considered as intermediate, in general features, between the "micro" methods of Folin and the standard methods employing a bank of block tin stills and necessary additional devices.

Continuance of the experimentation described by one of us[2] in this laboratory and in the Wooster laboratory gave rise to an apparatus which we feel merits recommendation to other workers in similar fields. Its features are: (1) elimination of rubber stoppers and connection; (2) efficient scrubbing of the entrained alkali from the steam; and (3) the use of Pyrex glass, which does not yield an appreciable amount of alkali to steam or boiling solutions.[3]

[1] Davis, A. R. A note on the adaptability of the Folin micro-Kjeldahl apparatus for plant work. Ann. Mo. Bot. Gard. **3**: 407–412. *pl.* 7. 1916.

[2] Allen, E. R. Some conditions affecting the growth and activities of Azotobacter chroococcum. Ann. Mo. Bot. Gard. **6**: 1–44. *pl. 1. f. 1–2.* 1919.

[3] Davisson, B. S. Ammonia and nitric nitrogen determinations in soil extracts and physiological solutions. Jour. Ind. and Eng. Chem. **10**: 600–605. *f. 1–3.* 1918.

The apparatus is shown in pl. 2, which is practically self-explanatory. All parts except the condenser jacket *G* are made from Pyrex glass, which may be worked with an oxy-illuminating gas flame. The bulb *A* is conveniently made from a 200-cc. flask, the neck being drawn down and sealed to the condenser tube *F*. The tip of the curved tube in bulb *A* is perforated by several holes at its lower point. Tube *C* is attached to the 500-cc. Kjeldahl flask *E* by a ground joint at *D*.

One objection to the apparatus in this form is its rigidity, which, on shaking to mix the alkali, in the Kjeldahl procedure, renders the likelihood of breakage high. To overcome this we have used a rubber joint at *B*, which does not appear to vitiate the results if the glass tubes are fitted closely end to end. An extreme case was taken to test this fault and the general efficiency of the apparatus. Solutions of N/100 acid and alkali were prepared and carefully standardized. A dilute solution of ammonium hydroxide was carefully titrated against the solutions, using methyl red as the indicator. Successive equal portions of this ammonium hydroxide solution were distilled in the apparatus and the distillate titrated with the above-described solutions. Among the following data those results indicated by an asterisk were obtained in an apparatus with a close rubber joint at *K*.

Nitrogen taken 0.103 mgs.

N found (mgs.)	Error (mgs.)
0.101	–0.002
0.105	0.002
0.103	0.000*
0.101	–0.002
0.098	–0.005*
0.103	0.000
0.099	0.004
0.098	–0.005*
0.105	0.002*
Average 0.102	Average deviation 0.0024

Scrubbing bulb *A* effectively removes the alkali entrained in the vapor when distillations are made over strongly alka-

line solutions. The surface of the bulb is large enough to provide sufficient condensation to form a water trap, through which fixed alkali will not pass. This point was tested by an experiment in which conditions were also purposely extreme. There were placed 100 cc. of concentrated alkali and 100 cc. of nitrogen-free water in the distilling flask and zinc added, just as in the case of the regular nitrogen determinations. The distillate was collected in three portions of about 80 cc. each, and in no fraction was there sufficient alkali to be detected with N/100 acid.

The requirements as to quality of joint at *D* are not so exacting as in the case of joints for ether extraction and similar apparatus, for the reason that a safe connection may be made with the aid of a water seal, using a joint less close-fitting than might otherwise be demanded. For this reason we hope the manufacturer will eventually be able to make the flasks interchangeable on different pieces of apparatus. This point is under consideration at the present time.

EXPLANATION OF PLATE

PLATE 2

An all-glass nitrogen apparatus. (See p. 46 for explanation.)